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SPECIFICATION FOR VULCANIZED FIBRE SHEETS FOR ELECTRICAL PURPOSES

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 7 *March* 1969

Indian Standard

SPECIFICATION FOR VULCANIZED FIBRE SHEETS FOR ELECTRICAL PURPOSES

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Indian Standard

SPECIFICATION FOR VULCANIZED FIBRE SHEETS FOR ELECTRICAL PURPOSES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 16 October 1968, after the draft finalized by the Insulating Materials Sectional Committee had been approved by the Electrotechnical Division Council.

0.2 Vulcanized fibre is made by combining layers of chemically gelled paper. The chemical compound used in gelling the paper is subsequently removed by leaching, and the resulting product, after being dried and finished by calendering, is a dense material of partially regenerated cellulose in which the fibrous structure is retained in varying degrees, depending upon the grade of fibre. It is made in four primary forms — sheets, rolls, rods and tubes.

0.3 This standard covers vulcanized fibre sheets of 1·5 to 25 mm thickness. Separate Indian Standard specifications deal with sheets of thickness less than 1·5 mm and with rods and tubes.

0.4 In damp conditions vulcanized fibre sheet is not a satisfactory electrical insulating material. It is recommended that when employed in dry conditions, vulcanized fibre sheet should not be subjected to a permanent working electric stress greater than 400 volts/mm.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard covers vulcanized fibre sheets of 1·5 to 25 mm thickness.

---

*Rules for rounding off numerical values (*revised*).

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Machine Direction — The direction parallel to that in which the material travelled during manufacture.

2.2 Cross Machine Direction — The direction at right angle to that in which the material travelled during manufacture.

2.3 Perpendicular Direction — The direction at right angle to the surface of the material.

3. CLASSIFICATION

3.1 Vulcanized fibre sheets shall be classified in the following two types:

a) *Type A* — A dense vulcanized fibre sheet having good mechanical and electrical properties.

b) *Type B* — Vulcanized fibre sheet having properties inferior to Type A.

4. REQUIREMENTS

4.1 Material — The material shall consist of superimposed layers of specially prepared paper chemically treated so that the laminæ are virtually destroyed with the production of a homogeneous mass of converted cellulose in the form of sheet. To confirm this conversion the material shall not delaminate after boiling in distilled water for 30 minutes. The vulcanized fibre sheet shall be free from deleterious substance.

4.2 Finish — The vulcanized fibre sheets shall be uniformly finished with a smooth mat surface and shall be free from twisting or splitting along the edge. The sheets shall be free from internal fractures.

4.3 Flatness — It shall be subject to agreement between the manufacturer and the supplier.

4.4 Preferred Thickness — The preferred thickness in millimetres shall be the following:

1·5, 2·0, 2·5, 3·0, 4·0, 5·0, 6·0, 7·0, 8·0, 9·0, 10·0, 12·5, 15·0, 17·5, 20·0, 22·5, 25·0

4.5 Tolerance on Thickness — The thickness of the sheets at any point when measured in accordance with Appendix A shall not differ from the specified nominal thickness by more than the appropriate tolerance giver in Table 1.

TABLE 1 TOLERANCE ON THICKNESS OF SHEETS

(*Clause* 4.5)

All dimensions in millimetres.

NOMINAL THICKNESS (1)	TOLERANCE (2)
1·5	± 0·12
2·0	± 0·23
2·5	± 0·23
3·0	± 0·23
4·0	± 0·30
5·0	± 0·30
6·0	± 0·30
7·0	± 0·38
8·0	± 0·38
9·0	± 0·38
10·0	± 0·63
12·5	± 0·80
15·0	± 0·80
17·5	± 0·80
20·0	± 1·00
22·5	± 1·00
25·0	± 1·00

4.6 Density — The minimum permissible density of the sheets, when determined by the method as described in Appendix B, shall be as shown in Table 2.

TABLE 2 DENSITY

NOMINAL THICKNESS mm	MINIMUM PERMISSIBLE DENSITY g/cm³	
	Type A	Type B
(1)	(2)	(3)
1·5	1·30	1·15
2·0	1·30	1·15
2·5	1·30	1·15
3·0	1·30	1·15
4·0	1·30	1·15
5·0	1·30	1·15
6·0	1·30	1·15
7·0	1·25	1·15
8·0	1·25	1·15
9·0	1·25	1·15
10·0	1·25	1·15
12·5	1·20	1·15
15·0	1·20	1·15
17·5	1·20	1·15
20·0	1·15	1·10
22·5	1·15	1·10
25·0	1·15	1·10

4.7 Electric Strength in Air at 90°C

4.7.1 *Proof Test* — When tested according to IS : 2584-1963*, the material shall withstand for one minute without failure and without any sign of steaming or smoking the appropriate voltage shown by the relevant curve in Fig. 1.

4.7.2 *Breakdown Test* — This test is applicable only for type A material. When tested as described in IS : 2584-1963*, the breakdown voltage of the material shall be not less than the appropriate value shown by the curve given in Fig. 2.

4.8 Shrinkage

4.8.1 *In Air* — When tested in accordance with Appendix C, the shrinkage shall not exceed the appropriate value given in Table 3 for all thicknesses of sheet.

4.8.2 *In Oil* — When tested in accordance with Appendix C, the shrinkage shall not exceed the appropriate values given in Table 3 for all thicknesses of sheet.

TABLE 3 SHRINKAGE OF SHEETS IN AIR AND IN OIL

Dimension	Maximum Permissible Shrinkage in Percentage	
	In Air	In Oil
(1)	(2)	(3)
Machine direction	2·0	2·0
Cross machine direction	3·0	3·0
Perpendicular direction	6·0	6·0

4.9 Delamination — After the tests in accordance with Appendix C, the material shall show no sign of delamination or cracking at the edges when examined with a hand lens (magnification of 5).

4.10 Tensile Strength — The tensile strength of vulcanized fibre sheet shall be not less than the appropriate value shown by the relevant curve in Fig. 3 and 4 when tested in accordance with Appendix D.

4.11 Shear Strength — The shearing strength of the material shall be not less than the appropriate value shown by the relevant curve in Fig. 5 and 6 when tested in accordance with Appendix E.

4.12 Flexibility — Sheets from 1·5 to 3·0 mm thick, when tested as described in Appendix F, shall show no sign of cracking or splitting when wrapped round a mandrel of the appropriate diameter given in Table 4.

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*Method of test for electric strength of solid insulating materials at power frequencies.

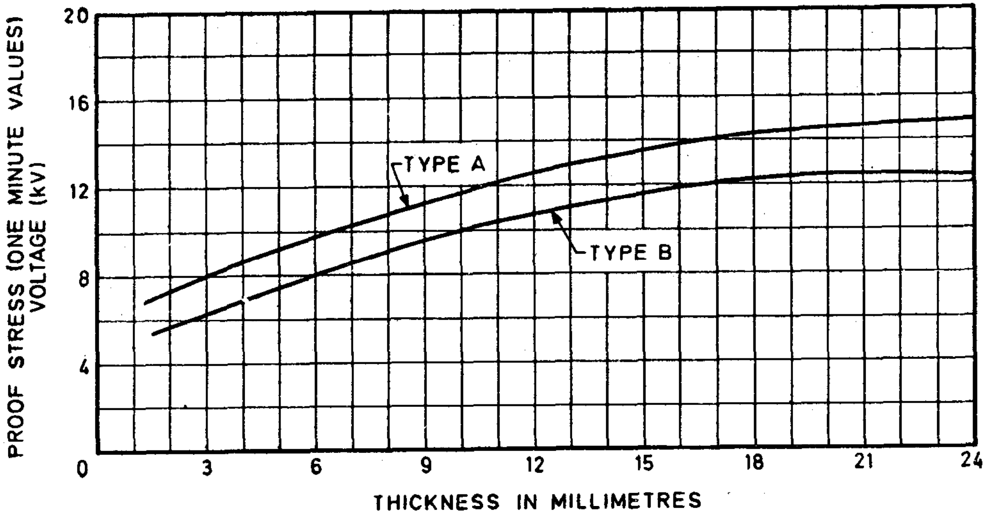

FIG. 1 ELECTRIC STRENGTH (PROOF TEST)

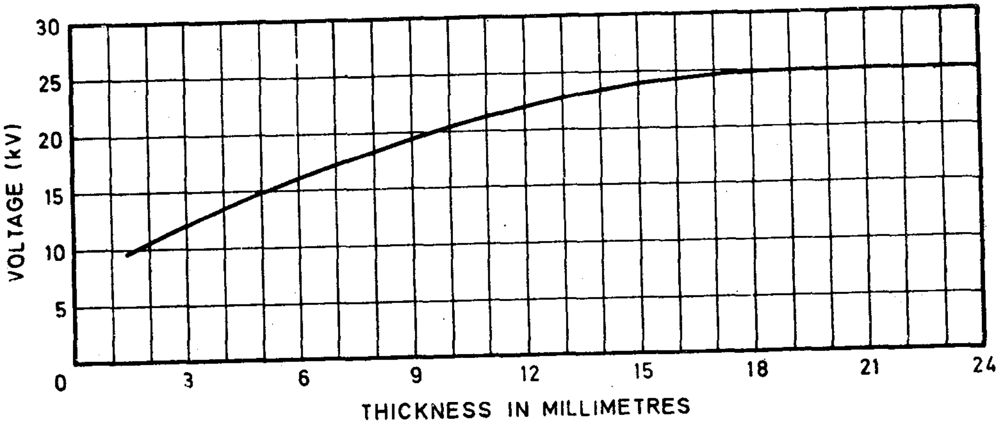

FIG. 2 MINIMUM BREAKDOWN STRENGTH — TYPE A MATERIAL

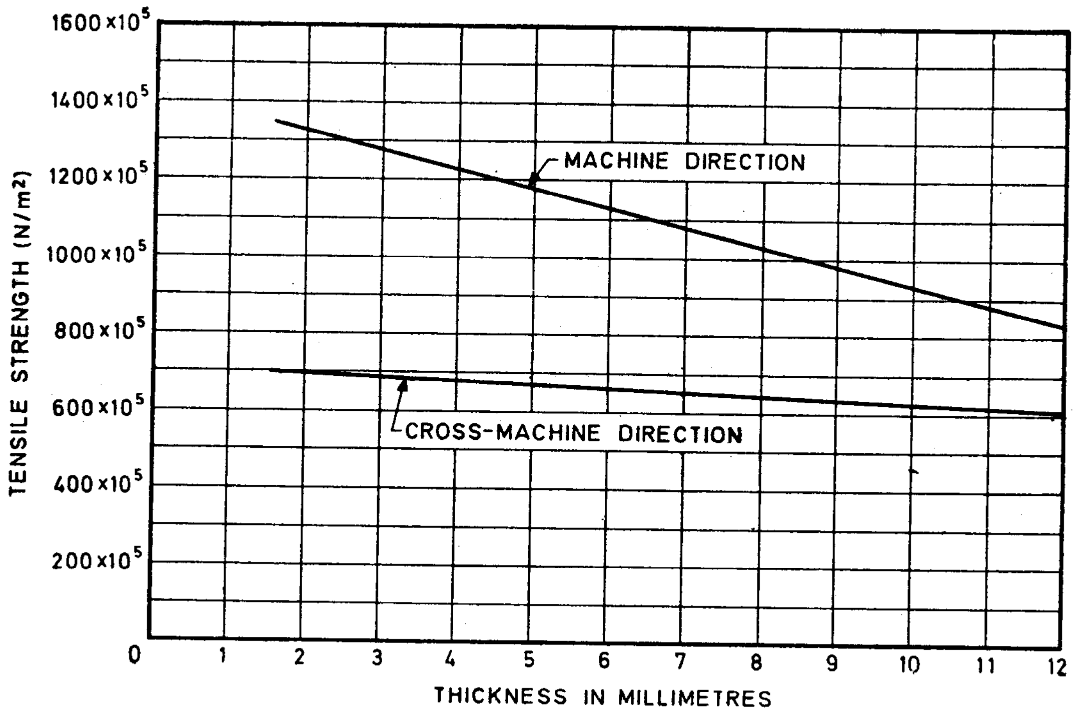

FIG. 3 TENSILE STRENGTH — TYPE A MATERIAL

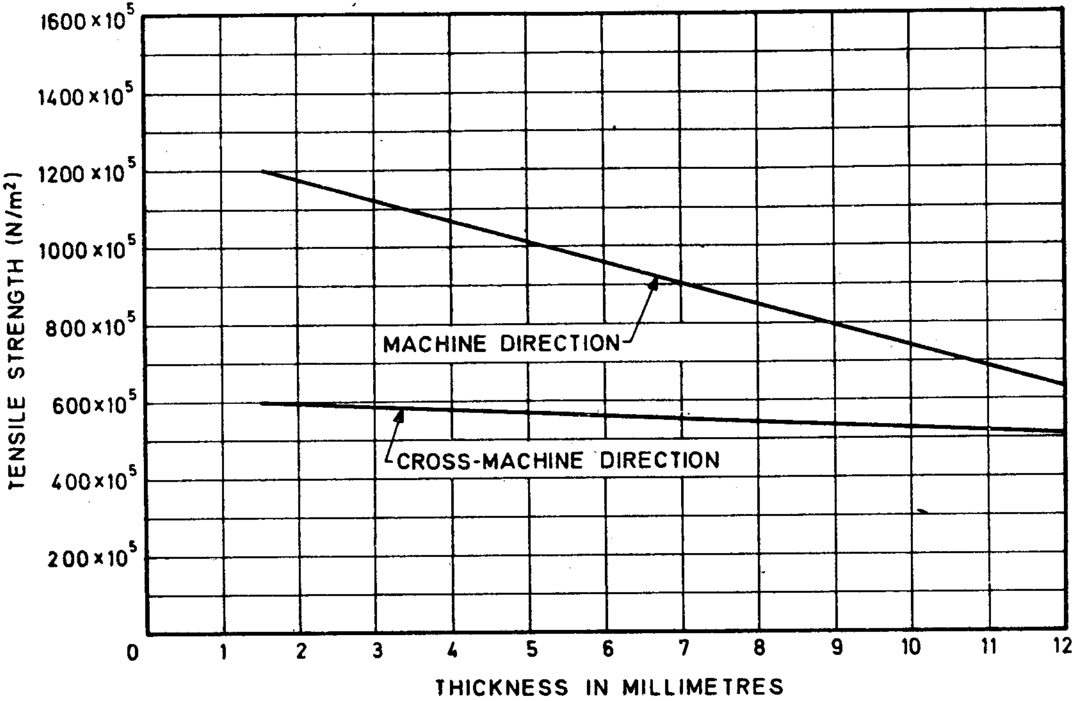

FIG. 4 TENSILE STRENGTH — TYPE B MATERIAL

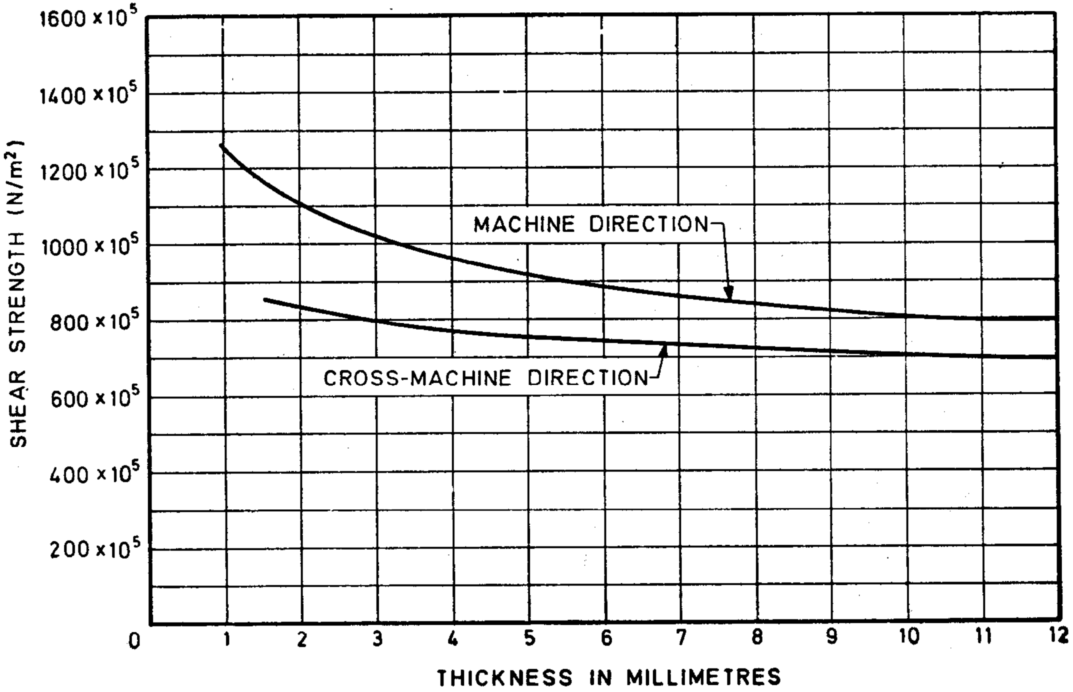

FIG. 5 SHEAR STRENGTH — TYPE A MATERIAL

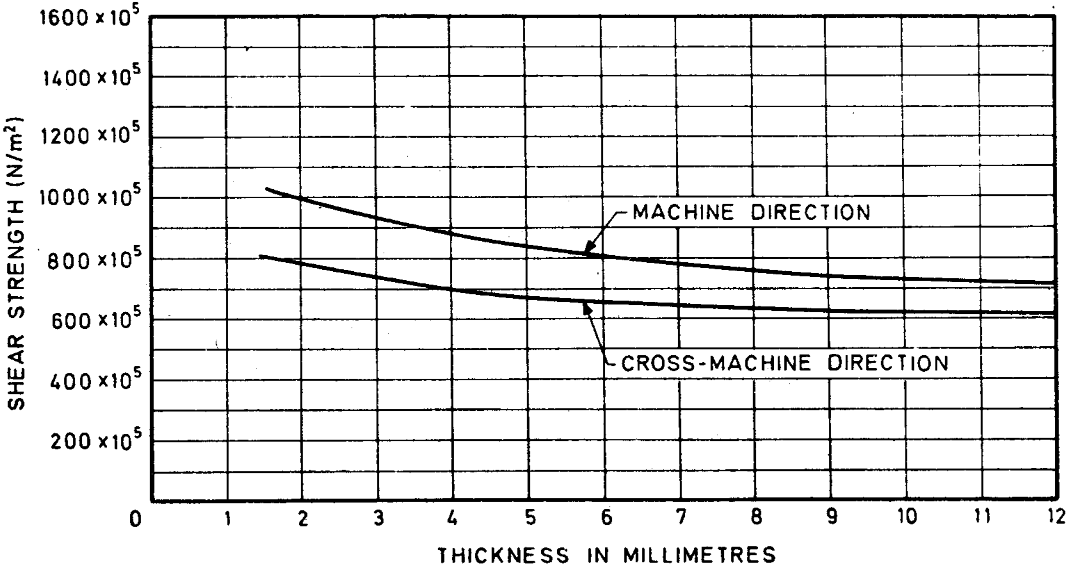

FIG. 6 SHEAR STRENGTH — TYPE B MATERIAL

TABLE 4 MANDREL DIAMETERS FOR FLEXIBILITY TEST

(*Clauses* 4.12 *and* F-2.1)

All dimensions in millimetres.

NOMINAL THICKNESS	DIAMETER OF MANDREL	
	Machine Direction Specimen	Cross Machine Direction Specimen
(1)	(2)	(3)
1·5 up to and including 2·5	19·0	22·0
Above 2·5 up to and including 3·0	44·0	57·0
Above 3·0	63·0	76·0

4.13 Bending (Proof) Test

4.13.1 Sheets above 3·0 mm in thickness, when tested in accordance with Appendix G, shall withstand for one minute without failure the appropriate proof load given in Table 5.

4.13.2 The deflection after the proof load given in Table 5 has been maintained for one minute shall be not less than 6·0 mm for a test piece cut in either the machine or cross machine direction of the sheet of any thickness.

TABLE 5 BENDING (PROOF) LOADS ON SHEETS

(*Clauses* 4.13.1, 4.13.2 *and* G-3.2)

NOMINAL THICKNESS OF SHEET	PROOF LOAD	
	Machine Direction Specimen	Cross Machine Direction Specimen
(1)	(2)	(3)
mm	kg	kg
Above 3·0 up to and including 4·5	45	35
,, 4·5 ,, ,, ,, 6·0	60	45
,, 6·0 ,, ,, ,, 12·0	180	135
,, 12·0 ,, ,, ,, 19·0	340	270
,, 19·0 ,, ,, ,, 25·0	450	360

4.14 Machinability — The sheet shall be capable of being sawn, milled, drilled, tapped and shaped in a shaping machine without showing any sign of splitting, cracking or chipping.

NOTE — All machining operations shall be carried out in accordance with the recommendations of the manufacturer.

4.15 Chloride Content — The total amount of chlorides, determined in accordance with Appendix H shall not exceed 0·05 percent up to and including 12·0 mm thickness and 0·1 percent above this thickness.

4.16 Sulphate Content — The total amount of sulphates, determined in accordance with Appendix J shall not exceed 0·05 percent.

4.17 Moisture Content — The moisture content of vulcanized fibre sheet shall not exceed 8 percent when tested in accordance with Appendix K.

4.18 Ash Content — The ash content shall not exceed 7 percent for red sheet and 3 percent for others when determined in accordance with Appendix L.

4.19 Silica Content — The silica content shall not exceed 0·3 percent when determined in accordance with Appendix M.

5. MARKING

5.1 The consignment of vulcanized fibre sheet shall be indelibly marked on a suitable place with the following information:

a) Manufacturer's name, trade-mark, if any;
b) Thickness;
c) Type A or B; and
d) Country of manufacture.

5.1.1 The vulcanized fibre sheet may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

6. SAMPLING AND CRITERION FOR CONFORMITY

6.0 For production control, use of control chart techniques is recommended to the manufacturer (*see* IS : 397-1952*). The results of such tests done at the place of manufacture may be made available to the purchaser to satisfy him that the material produced by the manufacturer is generally of the specified quality. In addition, the procedure specified in **6.1** to **6.4.4** may be adopted to ensure that the material conforms to this standard.

6.1 Lot — In any consignment all sheets of the same thickness manufactured under relatively similar conditions of production shall be grouped. Each lot shall be sampled separately for ascertaining the conformity to the requirements of the specification.

---

*Method for statistical quality control during production by the use of control chart (*tentative*). **(Since revised and split into various parts).**

6.2 Scale of Sampling — The number of sheets to be selected from a lot shall depend on the lot size and shall be in accordance with col 1 and 2 of Table 6.

6.2.1 The sheets shall be selected at random from the lot. Recommended procedure is given below:

> 'Arrange all the sheets in the lot in a systematic manner and starting from any sheet, count them as 1, 2, 3,...... up to *r* and so on, where *r* is the integral part of N/n, and N and n the lot size and sample size respectively. Every *r*th sheet thus counted shall be withdrawn to constitute the sample.'

TABLE 6 SCALE OF SAMPLING AND PERMISSIBLE NUMBER OF DEFECTIVES

(*Clauses* 6.2, 6.3.1 *and* 6.4.2)

LOT SIZE N (1)	SAMPLE SIZE n (2)	PERMISSIBLE NUMBER OF DEFECTIVES (3)
Up to 100	25	2
101 „ 200	30	3
201 „ 300	35	3
301 „ 500	40	4
501 and above	50	5

6.3 Number of Tests

6.3.1 All the sheets selected in accordance with Table 6 shall be inspected for appearance, flatness and thickness.

6.3.2 For the remaining characteristics five sheets shall be selected from the sample irrespective of the sample size. Tests shall be conducted on the specimens drawn from them.

6.3.3 While specimens are cut from the boards, ensure that no part of any test piece is from a position less than 25 mm from the edge of the board as received.

6.4 Criterion for Conformity

6.4.1 A lot shall be declared as conforming to the requirements of the specification, if the results of testing satisfy the conditions stipulated in **6.4.2** to **6.4.4**; otherwise, the lot shall be considered as not having met the requirements of the specification.

6.4.2 The number of sheets in the sample failing to meet the requirements for finish and tolerance on thickness shall be less than or equal to the corresponding permissible number of defectives given in col 3 of Table 6.

6.4.3 For tensile strength, shear strength, chloride and sulphate content and moisture content, the mean (X) and range (R) of the corresponding five test results shall be calculated (range is defined as the difference between the maximum and minimum value of the mean of the test results carried out on each sample sheet).

6.4.3.1 The conformity of the lot to the requirements for tensile strength, shear strength, shrinkage, deflection, chloride and sulphate content, moisture content, shall be declared only if the expression $X \pm 0{\cdot}4\ R$, as calculated from the relevant test results, is greater than or equal to the corresponding requirements.

6.4.4 For the remaining requirements, namely, density, electric strength, flexibility, delamination and bending the permissible number of defectives allowed in the five sheets selected for the purpose (*see* **6.3.2**) shall not be more than one.

APPENDIX A

(*Clause* 4.5)

METHOD FOR DETERMINATION OF THICKNESS

A-1. AS RECEIVED

A-1.1 To ascertain if the thickness of the sheet complies with the requirement of **4.5**, measurements of the thickness shall be made on the sheet in the condition as received, with a micrometer conforming to IS : 2967-1964*, at 10 points equally spaced around the four sides of the sheet of vulcanized fibre or at equal distances across the width.

A-2. AFTER NORMAL CONDITIONING

A-2.1 If the purchaser is doubtful as to whether the thickness conforms to the requirements given in **4.5**, a strip 40 mm wide shall be cut from one end of the sheet and along the entire cross machine direction. Ten specimens each not less than 40 mm long shall be cut from ten positions equally spaced along the strip. The specimens shall be conditioned as described in Appendix N, and the thickness of each specimen shall then be measured with a micrometer conforming to IS : 2967-1964*, one measurement being made near the centre of each specimen. The mean thickness computed from these measurements shall be deemed to be the thickness of the board.

---

*Specification for external micrometers.

APPENDIX B

(*Clause* 4.6)

METHOD FOR DETERMINATION OF DENSITY

B-1. The specimen shall be 40 × 40 mm. Condition the specimen as described in Appendix N. The weight of the specimen shall be ascertained to the nearest milligram. The volume in cubic centimetres shall be ascertained by weighing the specimen in air and in toluole of known specific gravity and care shall be taken to obtain the weight of the specimen in the liquid as rapidly as possible so as to minimize the error due to the absorption of the liquid by the specimen. The density of the specimen shall be computed as follows:

$$\text{Density of the specimen} = \frac{W_1 \times \sigma}{(W_1 - W_2)} \text{ grams per cubic centimetre}$$

where

W_1 = weight in grams of specimen in air, and

W_2 = weight in grams of specimen in the toluole of specific gravity σ.

NOTE — When acceptance tests are being made on a bulk consignment of vulcanized fibre sheet, it is desirable to weigh and measure the entire sheets as received. This test affords a rapid means of checking the density of the material without cutting the sheet, and should be conducted before cutting specimens for other tests.

It should be appreciated that the results obtained by this rapid method are lia be 10 to 20 percent lower than those obtained by the method described in the body o the Appendix, which shall be used in case of dispute.

APPENDIX C

(*Clauses* 4.8.1, 4.8.2 *and* 4.9)

SHRINKAGE

C-1. TEST PIECE

C-1.1 The test piece shall be 40 mm square. No part of the specimen shall be cut from a position nearer than 25 mm from an edge of the sheet as received.

C-2. CONDITIONING

C-2.1 Condition the test piece in accordance with Appendix N.

C-3. PROCEDURE

C-3.1 The dimensions of the specimen shall be measured accurately by means of a micrometer or other suitable instrument. The length and width respectively of the specimen shall be the mean of three measurements taken at points equally spaced along each of two edges at right angles. The thickness of the specimen shall be the mean of four measurements of thickness one being taken adjacent to each side of the specimen.

C-3.1.1 *Shrinkage in Air* — The specimen shall be heated in a well ventilated oven for 48 hours at a temperature of 105° ± 2°C. Immediately measure the dimensions as before.

C-3.1.2 *Shrinkage in Oil* — After the dimensions of the specimen are determined as in **C-3.1**, heat the specimen in a well ventilated oven for 48 hours in insulating oil complying with IS : 335-1963* at a temperature of 105° ± 2°C. On removal of the test piece from the oil wipe off excess oil and after allowing it to cool to room temperature measure the dimensions as before.

C-4. RESULTS

C-4.1 Calculate the shrinkage in air as the percentage change in dimensions of the original measurements of the conditioned test piece.

C-4.2 Calculate the shrinkage in oil as the percentage change in dimensions or the original measurements of the conditioned test piece.

APPENDIX D

(*Clauses* 4.10, F-1.1 *and* G-1.1)

TENSILE STRENGTH

D-1. TEST PIECE

D-1.1 The form and dimensions of the specimen for test shall be as shown in Fig. 7, or alternatively use strips of uniform width from 12·5 to 25·0 mm. The thickness of the specimen shall be the thickness of the sheet under test. Sheets above 12·0 mm thick may be machined down to 12·0 mm thickness. The free length of each test piece between the jaws of the testing machine shall be not less than 150 mm.

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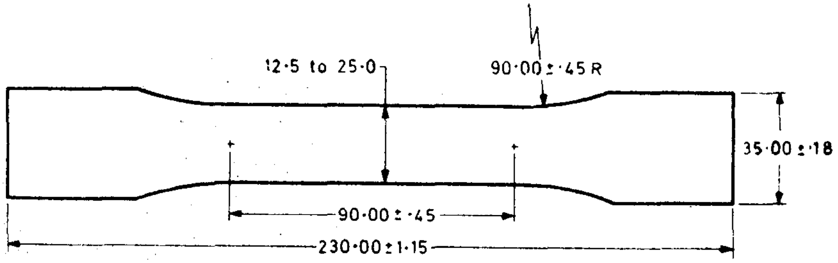
*Specification for insulating oil for transformers and switchgear (*revised*). (Since revised).



All dimensions in millimetres.

FIG. 7 SPECIMEN FOR TENSILE STRENGTH TEST

D-1.2 Use three test pieces cut in each machine and cross machine directions. In cutting test pieces from the sheet under test no part of any test piece shall be cut from a position nearer than 25 mm from the edge of the board as received.

D-1.3 If the machine and cross machine directions are not known, cut a test piece with its length in a direction parallel to one side of the sheet and another in the direction at right angles (*see* Fig. 8). The test piece having the greater value of tensile strength is deemed to have been cut with its length in the machine direction of the sheet.

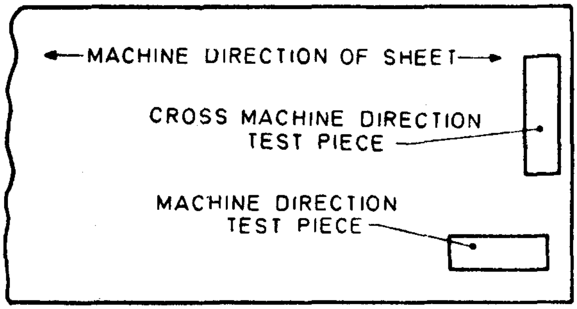

MACHINE DIRECTION OF SHEET
CROSS MACHINE DIRECTION TEST PIECE
MACHINE DIRECTION TEST PIECE

FIG. 8 DIRECTIONS IN WHICH TEST PIECES ARE CUT FROM SHEETS

D-2. CONDITIONING

D-2.1 Condition the test pieces in accordance with Appendix N.

D-3. PROCEDURE

D-3.1 Measure the thickness and width of each test piece on removal from the conditioning atmosphere. Make the test at a temperature of 10° to 40°C.

Apply the load steady so that the time taken to reach the breaking load from the commencement of application of the load is not less than 30 seconds. Disregard the results of test pieces breaking within the jaws.

D-4. RESULTS

D-4.1 Report the machine and cross machine tensile strength in N/m² calculated as the mean of the results from the three test pieces cut in each direction.

APPENDIX E

(*Clause* 4.11)

SHEAR STRENGTH

E-1. TEST PIECE

E-1.1 The specimen shall be smoothly cut and shall be of the following dimensions:

Length 95·0 mm and width 20·0 mm (± 0·25 mm).

The thickness of the specimen shall be the thickness of the sheet under test. Sheets above 12·0 mm thick may be machined down to 12·0 mm thickness.

E-1.2 Use three test pieces cut in each machine and cross machine directions. In cutting test pieces from the sheet under test no part of any test piece shall be cut from a position nearer than 25 mm from the edge of the sheet as received.

E-1.3 If the machine and cross machine directions are not known cut a test piece with its length in a direction parallel to one side of the sheet and another in the direction at right angles to this. The specimen having the greater value of shear strength shall be deemed to have been cut with its length in the machine direction of the sheet or roll.

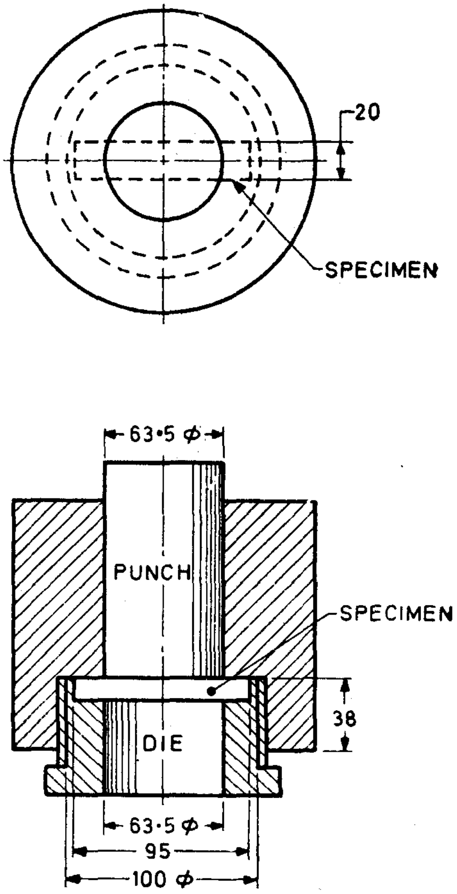
E-2. CONDITIONING

E-2.1 Condition the test piece in accordance with Appendix N.

E-3. PROCEDURE

E-3.1 Measure the thickness and width of each test piece on removal from the conditioning atmosphere. Make the test at a temperature of 10° to 40°C.

E-3.2 The tests are carried out by means of a circular punch and die of the dimensions shown in Fig. 9. The punch shall be a running fit in the die and shall not weigh more than 10 percent of the minimum shear load specified. The load shall be applied axially to the specimen, which shall be securely clamped in place and the time taken to reach the breaking load from the commencement of the application of the load shall be appropriate to the type of testing apparatus used and in any case shall be approximately two minutes.



All dimensions in millimetres.

FIG. 9 PUNCH AND DIE FOR SHEAR STRENGTH TEST

E-4. RESULTS

E-4.1 Shear stress shall be calculated by the following formula:

$$S = \frac{W}{2\,BT}$$

where

S = shear stress in N/m^2,
W = shearing load in Newton,
B = width of specimen in m, and
T = thickness of specimen in m.

E-4.2 Report the machine and cross machine direction shear strength calculated as the mean of the results from the three test pieces cut in each direction.

APPENDIX F

(*Clause* 4.12)

FLEXIBILITY

F-1. TEST PIECES

F-1.1 Three test pieces shall be cut 25 mm wide and 300 mm long from both the machine and cross machine directions of the sheet. In cutting these test pieces ensure that no part of any test piece is from a position less than 25 mm from the edge of the sheet as received.

NOTE — If the machine and cross machine directions are not known they may be ascertained from the result of tensile strength test (*see* Appendix D).

F-2. CONDITIONING

F-2.1 The specimens shall be heated in an oven for 48 hours at a temperature of 105° ± 2°C and allowed to cool in a controlled atmosphere as described in Appendix N. They shall be maintained at this atmosphere for 18 to 24 hours. Each specimen shall then be bent through an angle of 90° round a mandrel of the appropriate diameter given in Table 4. The rate of bending being such that the complete bend is performed in two minutes, the effect on the specimen shall be noted.

APPENDIX G

(*Clause* 4.13.1)

BENDING (PROOF) TEST

G-1. TEST PIECE

G-1.0 This test is applicable only to sheets whose thicknesses lie between 3·0 mm and 25·0 mm.

G-1.1 Cut a test piece 25 mm wide and of suitable length from the machine direction of the sheet and a similar from cross machine direction of the sheet. In cutting these test pieces ensure that no part of the test piece is from a position less than 25 mm from the edge of the sheet as received.

NOTE — If the machine and cross machine directions are not known, they may be ascertained from the results of the tensile strength test (*see* Appendix D).

G-2. CONDITIONING

G-2.1 Heat the test pieces in an oven for 48 hours at a temperature of 105° ± 2°C and allow it to cool in a controlled atmosphere of RH 65 ± 2 percent and temperature 20° ± 2°C (*see* Appendix N) and maintain it for 18 to 24 hours.

G-3. PROCEDURE

G-3.1 After conditioning as in **G-2.1** support the test piece on two rods each 9·5 mm diameter spaced apart at a distance between centres as shown in Table 7.

TABLE 7 SPACING OF RODS BETWEEN CENTRES FOR BENDING TEST

All dimensions in millimetres.

NOMINAL THICKNESS (1)	SPACING OF RODS BETWEEN CENTRES (2)
Above 3·0 up to and including 4·5	108
,, 4·5 ,, ,, ,, 6·0	127
,, 6·0 ,, ,, ,, 9·0	152
,, 9·0 ,, ,, ,, 12·0	178
,, 12·0 ,, ,, ,, 16·0	203
,, 16·0 ,, ,, ,, 19·0	216
,, 19·0 ,, ,, ,, 22·0	235
,, 22·0 ,, ,, ,, 25·0	254

G-3.2 Place the test piece on the rods with its sheet surfaces horizontal. By means of a rod 9·5 mm diameter apply a load to the upper surface of test piece at a position centrally between the two supports. Increase the load at a steady rate, between 159-204 kg/min, until the appropriate load given in Table 5 is reached.

G-3.3 Maintain this load for one minute and using a micrometer or other suitable instrument, immediately measure the deflection at the centre of the span.

APPENDIX H

(*Clauses* 4.15 *and* J-2.1)

CHLORIDE CONTENT

H-1. PRECAUTIONS

H-1.1 In storage of samples of vulcanized fibre sheets for tests for chlorides content, adequate care should be taken to see that they are not contaminated by the atmosphere particularly the atmosphere of a chemical laboratory.

H-2. PREPARATION OF AQUEOUS EXTRACT

H-2.1 The material shall be cut into pieces not exceeding 25·0 mm in length and 3·0 mm in width. Twenty grams of the pieces shall be boiled in 300 ml of distilled water for one hour. The extract shall be filtered, the filtering medium washed and the washings added to the filtrate. Excess precipitated chalk to neutralize the solution shall be added.

The neutralized solution shall then be boiled for ten minutes and filtered. The solution shall be boiled down to between 100 and 150 ml and, if necessary, filtered again.

H-3. TITRATION OF AQUEOUS EXTRACT

H-3.1 Normal Method — This method is applicable when the solution prepared by the above method is clear and free from colour. When cold, the solution shall be titrated with standard decinormal silver nitrate solution, using potassium chromate as an indicator.

The result shall be reported as a percentage by weight of the chloride ion on the original weight of the specimen taken for test.

H-3.2 Potentiometric Method

H-3.2.1 This method is applicable when coloration of the material comes out in the extract, or the extract is cloudy.

H-3.2.2 *Apparatus* — The apparatus consists of two stout pure silver wires dipped into two small beakers containing:

a) the solution under test (in convenient quantity, accurately measured) acidified with one drop of 25 percent nitric acid.

b) a saturated solution of silver nitrate acidified with one drop of 25 percent nitric acid, and also containing 1 to 2 ml of molar ammonium nitrate solution.

Electrical connection between the two beakers shall be made by means of a U-tube filled with molar ammonium nitrate solution and plugged at each end with filter paper.

H-3.2.3 Titration shall be carried out with 0·002 N silver nitrate solution and the change in emf measured by means of a valve-potentiometer, the solution being stirred continuously. The emf of the cell shall be plotted against the volume of standard silver nitrate added. The end point shall be the point of inflection, which may be most easily found by plotting the values of $\Delta E/\Delta V$ (where ΔE is a small change in emf corresponding to a small change in volume ΔV) against the volume of titrating solution. At the point of inflection the value of $\Delta E/\Delta V$ is a maximum. The result shall be reported as a percentage by weight of chloride ion on the original weight of the specimen taken for test.

APPENDIX J

(*Clause* 4.16)

SULPHATE CONTENT

J-1. PRECAUTIONS

J-1.1 In the storage of vulcanized fibre sheets intended for test for sulphate content adequate care should be taken to ensure that they are not contaminated by the atmosphere particularly the atmosphere of a chemical laboratory.

J-2. EXTRACTION

J-2.1 Fifty millilitres of an extract prepared as in Appendix H shall be used for this test.

J-3. DETERMINATION

J-3.1 The extract shall be placed in a clean boiled out resistance-glass beaker and 1 to 2 ml of 10 percent dilute hydrochloric acid is added. The solution shall be brought to boiling point and 1 to 2 ml of 5 percent barium chloride solution added. Boiling shall be continued for five minutes and the solution left to cool and stand overnight. Any precipitate of barium sulphate which forms shall be filtered, washed free from chlorides, dried and ignited. It shall then be moistened with concentrated sulphuric acid, re-ignited and weighed.

J-4. RESULTS

J-4.1 The weight of sulphate = Weight of barium sulphate × 0·412

$$\text{Sulphate content of specimen} = \frac{\text{Weight of sulphate}}{\text{Weight of original material}} \times 100$$

APPENDIX K

(*Clause* 4.17)

MOISTURE CONTENT

K-1. SPECIMEN

K-1.1 The specimen shall be approximately 5 g minimum weighed to the nearest milligram.

K-2. PROCEDURE

K-2.1 The specimen shall then be dried to constant weight by heating in a well-ventilated oven at a temperature of 105° ± 2°C, the usual precaution being taken.

K-3. MOISTURE DETERMINATION

K-3.1 The moisture content shall be determined from the difference between the weight of the specimen in the conditions as received and after the complete drying to constant weight.

K-4. RESULTS

K-4.1 The percentage of moisture in the vulcanized fibre shall be computed on the dry weight of the specimen.

APPENDIX L

(*Clause* 4.18)

ASH CONTENT

L-1. TEST SPECIMEN

L-1.1 The test specimen shall consist of 2 to 5 g of finely divided particles, such as millings or filings of the material.

L-2. PROCEDURE

L-2.1 Dry the test specimen for 2 hours at 105° to 110°C and weigh. Then ignite the specimen to constant weight in a crucible and re-weigh. Calculate the percentage of ash, based on the weight of the dried specimen.

APPENDIX M

(*Clause* 4.19)

SILICA CONTENT

M-1. TEST SPECIMEN

M-1.1 The test specimen shall consist of approximately 3 g of finely devided particles, such as millings or filings of the vulcanized fibre sheet.

M-2. PROCEDURE

M-2.1 Dry the test specimen for one hour at 105° to 110°C. Transfer the dried specimen to a crucible and slowly ignite it to constant weight. Wet the ash with distilled water and transfer to a heat-resistant glass beaker. Add about 75 ml of HCl (sp gr 1·19) and cover the beaker with a watch glass. Evaporate the contents of the beaker to dryness. To the residue slowly add 10 ml of HCl (sp gr 1·19) followed by 75 ml of distilled water. Filter the mixture through ashless filter paper and wash with cold water, then with warm water, until the filtrate is free from chlorides. Ignite the filter paper to constant weight in a weighed platinum crucible. Then add 4 ml of HF (48 to 60 percent) and apply heat until all white fumes are driven off. Cool and weigh the crucible. The difference between this weight and the previous one indicates silicon present as silica. The results shall be expressed as a percentage of the weight of the dried specimen.

APPENDIX N

(*Clauses* A-2.1, B-1, C-2.1, D-2.1, E-2.1, F-2.1 *and* G-2.1)

CONDITIONING OF TEST PIECES

N-1. Condition the test pieces for not less than 18 hours in a controlled atmosphere of 65 ± 2 percent RH at a temperature of 27° ± 2°C. Commence the relevant test within three minutes of removal of each test piece from the controlled atmosphere.

N-2. This atmosphere of 65 percent RH may be obtained conveniently in an enclosed chamber in which a saturated solution of sodium nitrite is exposed to the atmosphere in the chamber. Prepare the saturated solution from distilled water and sodium nitrite of a recognized analytical reagent quality.

N-3. Expose the saturated solution so that the maximum surface is in contact with the air in the chamber, for example, by covering the floor of the chamber with a tray containing the saturated solution. It is essential to use an ample excess of solid sodium nitrite in contact with the saturated solution. If desired, a mobile slurry of salt and saturated solution may be used, this being prepared by adding to the solid salt, of suitable particle size, a previously prepared solution of sodium nitrite until the desired fluid consistency is obtained.

N-4. The saturated salt solution of slurry shall become contaminated in course of time; the salt tray should, therefore, be emptied, washed all over, carefully dried and refilled with sufficient saturated solution or slurry at intervals of not more than two months. More frequent charges may be necessary if the chamber is used where conditions of severe contamination are experienced. Ensure uniform conditions throughout the chamber through the passage of a fan circulating air over the surface of the saturated solution or slurry and around the test pieces. Free access of the conditioning atmosphere to all test pieces is essential. From time to time check that the humidity of the air in the chamber is correct.

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